RESEARCH ARTICLE

Mechanical Properties of novel hardened cement paste reinforced with Multi-Walled Carbon Nano-Tubes (MWCNTs) and Glass Fibers Nano material

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ABSTRACT: To improve the mechanical and durability properties of ordinary Portland cement (OPC) mortar and paste, the incorporation of multi walled Carbon nanotubes (MWCNTs) and their dispersion procedures, functionalization, and ultra sonication have been intensively implemented. Most of the studies showed significant enhancements in the mechanical properties of OPC mortar or paste; however, others showed impairments. The recent studies regarding the implementation of MWCNTs and Glass Fibres on the mechanical properties of OPC paste and mortar were reviewed and these properties include compressive, tensile, flexural strengths, and elastic modulus. A statistical study was conducted to evaluate the mechanical properties of concrete by dispersion of MWCNT's and Glass Fibres in the cement paste. In these composites, the percentage of MWCNTs was fixed at 0.75% by weight of cement, while the percentage of Glass Fibers was fixed at 0.25% by weight of cement. The samples were cured in tap water for 28 days at 25 + 2°C.Composite specimens were tested for compression and flexure in order to evaluate their mechanical properties such as compressive strength, flexural strength, toughness and ductility and compared with the results of plain cement control beams. The maximum deflection was found to be 0.5mm with a maximum load of 500N. The flexural strength was observed to be 1250.50 N/mm2 as per ASTM D 790 which is 20% more than the flexural strength obtained with Plain Cement+MWCNT's and 60 to 70% more than that obtained with Plain Cement + Glass Fibres. The flexural modulus as per deflection criteria is 535.94 N/mm2 which is 10 to 20% more than that obtained of Plain Cement+MWCNT's and Plain Cement+ Glass Fibres. The compressive strength of Plain Cement+0.75% MWCNT's+0.25% Glass fibres was found to be 65 N/mm2 which is greater than Plain cement and Plain cement+MWCNT's. Surface morphology by Scanning Electron microscopy of the specimens infers the clustering of glass fibres and demonstrating the dispersion and interaction between the MWCNTs and Cement paste.

KEYWORDS: Carbon fiber; Multi walled carbon nanotubes; Polymer matrix; Flexural strength; Mechanical properties

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INTRODUCTION

Nano materials have been proposed as reinforcement for cement matrix as they exhibit extraordinary strength and mechanical properties with the advancement in nanotechnology. These nano materials showed excellent resistance against the propagation of cracks in the cement matrix at the nano scale level and micro level and essentially created a new generation of cement composites having remarkable energy absorption capacity before failure.

To date, several nano materials have been utilized to reinforce cement paste, mortar and concrete, for instance, carbon nano fibers [1], carbon nanotubes [2,3], graphene [4], Nano sized titanium dioxide (TiO2) [5], nano silica [6], and nano clay [7].

Cement is the principal binder holding the aggregates together to produce concrete in the presence of water for hydration. As an engineered material, concrete composites are desired for their excellent compressive strength. However, the major disadvantage of concrete is its brittle nature which is attributed to its poor resistance to crack formation, low tensile strength and strain capacities. [8]

The engineering properties of CNT–OPC pastes were investigated with varied ultrasonication energy (UE) and CNT concentration. It was found that UE could effectively improve the aqueous dispersion of surface functionalized. A PC to CNTs mass ratio of 8 is recommended for ensuring effective dispersion of CNTs and maintaining workability of CNT–OPC pastes under sufficient ultrasonication. [9]

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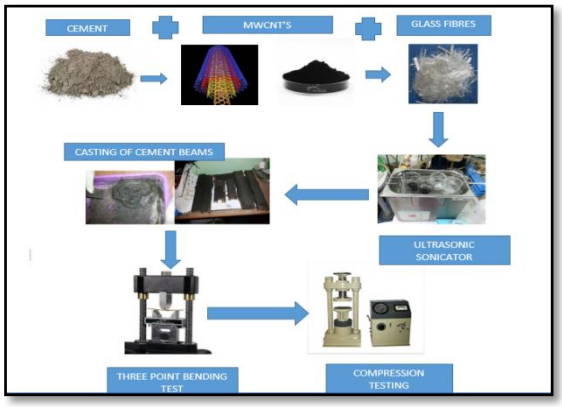
Carbon nanotubes have many advantageous mechanical and electrical properties such as high strength, high conductivity and, therefore, are attractive for producing fiber-reinforced concrete. Moreover, the incorporation of fibres at the nanoscale will allow the control of the matrix cracks at the nanoscale level. Mechanically, CNTs show elastic behavior, with a Young's Modulus of approximately 1 TPa and a density of about 1.33 g/cm3. Single walled CNTs have yield stresses between 20 and 60 GPa, with measured yield strains of up to 10% [10].

Since CNTs exhibit great mechanical properties along with extremely high aspect ratios (length-to-diameter ratio) ranging from 30 to more than many thousands, they are expected to produce significantly stronger and tougher cement composites than traditional reinforcing materials (e.g. glass fibers or carbon fibers). In fact, because of their size (ranging from 1 nm to tens of nm) and aspect ratios, CNTs can be distributed in a much finer scale than common fibers, giving as a result a more efficient crack bridging at the very preliminary stage of crack propagation within composites [10]

The aim of this paper is to study the mechanical properties of composites such as compressive strength, flexural strength, flexural modulus, toughness, ductility etc and the effect of nano-based additives on the overall performance of such composites. The main objective of this research paper is to reinforce cement matrix at nano-level by incorporating MWCNTs and at micro level by Glass Fibers and investigate the changes in mechanical properties of resulting composites.

EXPERIMENTAL METHOD

Fig-1. Depicting the Experimental Program



Materials

Cement (Ordinary Portland Cement), Multiwalled Carbon Nano tubes (MWCNT's) whose diameter vary from 4 to 30 nm and length varies from 1-10 micrometers possessing carboxyl and hydroxyl functional groups in the form of powder. The strength of the carbon-carbon bond is among the highest and as a result any structure based on aligned carbon-carbon bonds will have the ultimate strength. Carbon nano-tubes are therefore the ultimate high strength fibers. The Young's Modulus for MWCNT is 1.28 TPa, Glass Fibres in the form of thin strands purchased from Sigma Aldrich USA, the flexural strength is improved by the use of glass fibers due to their high tensile strength of 1.700 - 3.700 N/mm2 and their good bond with the cement matrix. This effect is seen only when the fibre content ranges from 3% - 5% of concrete volume [30].Water for mixing, moulds to prepare cement beams.

Preparation of Mix

MWCNTs in the form of powder were placed in a glass beaker. These nanotubes were pre dispersed in water by adding the 1/2 of the total water, which was used for the preparation of samples, was added to ensure that they were immersed completely into water. The beaker was vibrated by ultrasonic sonicator. Sonication was carried for 15 minutes and after that, the dispersant Ethanol of quantity 5 % by weight of cement was added to the pre dispersed mixture and again sonicated for another 15 minutes with continuous stirring. Addition of ethanol acts as a pre dispersing agent and thus bundled nano tubes gets dispersed.

The glass fibers cannot be dispersed by this method of sonication and hence the glass fibers were first immersed in the water and the fibers were separated by using a stirring rod and then the 0.25% of glass fibers by weight of cement was added to the cement and mixed manually by a desired water content.

First the experiment was conducted using only the varied percentage of glass fibers i.e 0.25%, 0.5%, 0.75% and 1% by weight of cement and the cubes were casted to know the optimum dose of glass fibers to be used for actually conducting the test. Specimens of 20mm×20mm×80mm were tested at the age of 28 days, by three point bending test. After knowing the optimum dosage of glass fibers by load deflection values, that specimen which gave the maximum deflection and load was taken as the optimum dosage of glass fibers. The one giving maximum deflection and maximum load is the specimen with the corresponding percentage of glass fibers and hence in the study the optimum percentage was found to be 0.25%.

Later the percentage of MWCNTs was fixed as 0.75% by the study of literature of various authors which gave optimum result. [31]. Experimental program is as shown in Fig-1.

The cubes were cast of size 20mm×20mm×80mm. Coromandel 43 grade OPC cement was used in the present investigation. It was tested as per IS-4031-1988 recommendation for hydraulic cement.

Specimens of 20mm×20mm×80mm were tested at the age of 28 days, by three point bending test. The test was performed with a load frame testing machine with a 10 kN capacity and with strain rate of 0.125mm/min, the flexural deformations of the beam specimen and its corresponding load are noted down.

Three point flexure tests on 20mm×20mm×80mm beams were carried out to compare the flexural strength of cement beams and composite beams. Later compressive test was carried out on the composite cubes to check the compressive strength, toughness and ductility.

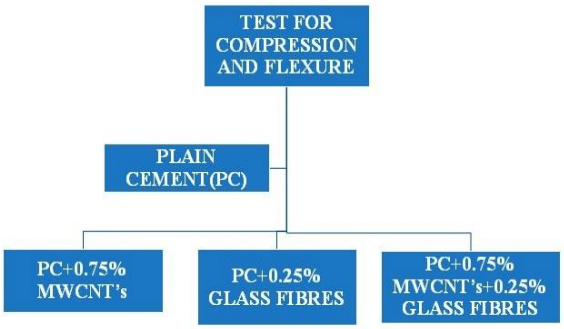


Figure-2. Depicting the composition of Cement Composites

Table 1. Details of test specimen

Specimen	Composition
PC	Plaincement
A1	Plaincement+0.75%MWCNTs
A2	Plaincement+0.25% Glass Fibers(GFs)
A3	Plaincement+0.75%MWCNTs+ 0.25%GFs

Characterizations

The surface morphology was evaluated using Field Emission Scanning Electron Microscopy to know the amount of elements present in the composite materials.

RESULTS AND DISCUSSION

Variation of flexural behavior of composites:

The Figure-3 shows the variation of modulas of Rupture and also the displacement at failure for all compositions of cement composites. It is clear from the graph that for Sample A3 as shown in Table 1 which is the combination of Plain Cement+0.75% MWCNT's+0.25% Glass Fibres shows the failure at ultimate load which means that the load carrying capacity of A3 increases.

The length of the fibres and specific area of the composites affects the strength in a much greater way as the aspect ratio and length of fibres help in bridging the cracks in the cement composite and they help in increasing the flexural strength of the composite material. As there is a change in the proportions of composites, the load carrying capacity has increased and the corresponding deflection has also increased. However the specimens with MWCNTs are found to be stiffer than cement paste and Glass fibres as evident from Fig-3.

A significant increase in the tensile/flexural strength of the composite is observed and considerable increase in the toughness of the composite is obtained.

In accordance with the expectations, the load deflection behavior showed that there was a remarkable increase in both flexural strength and toughness due to addition of nanoparticles that behave differently. The load carrying capacity was enhanced by about 21% by the addition of 0.75wt% of MWCNTs to the cement paste.

The Fig-5 represents or depicts the failure load of different compositions which helps in knowing the amount of load at which the specimen fails. It is clear from the graph that the A3 composite (PC+ 0.75% MWCNT's+0.25% Glass Fibres) shows the failure at maximum load. It is understood that the strength is considerably increased whereas for other combinations it is less.

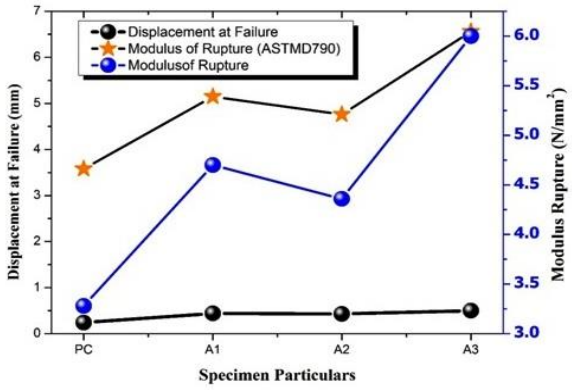


Figure-3. Depicting the variation of modulas of rupture for all compositions

Table 2. Table depicting variation in average values of mechanical properties for Cement composites under three point loading

Sl No	Specimen Particulars	Failure Load (kN)	Displacement at Failure Δmax (mm)	Modulas of Rupture (N/mm2)	Modulas of Rupture (ASTM D790)	Flexural Modulas (using deflection criteria)	Flexural Modulas (ASTM D790)
1	PC	273	0.24	3.28	3.58	609.62	480.5
2	A1	392	0.44	4.7	5.15	477.47	1060.8
3	A2	363	0.43	4.36	4.76	452.43	350.25
4	A3	500	0.5	6	6.56	535.94	1250.5

Table 3. Table depicting variation in Compressive strength

Sl No	Particulars of specimen	Compressive Strength N/mm²
1	**PC-** Plain cement	42
2	**A1-** Plain cement + 0.75% MWCNTs	56
3	**A2-** Plain cement +0.25 %Glass Fibers	55
4	**A3-** Plain cement + 0.75% MWCNTs+ 0.25%Glass Fibres	65

The Fig-6 shows the graph of Toughness index of all combinations of composites. Again from the graph it is clear that the toughness index for A3 (PC+ 0.75% MWCNT's+0.25% Glass Fibres) composite is high. Toughness is characterized generally as the amount of damage a material can withstand before fracture. The Fig-7 represents or depicts the Ductility Index of different combination of composites which again shows the A3 composite is having high ductility which is the degree to which a material can sustain plastic deformation under tensile stress before failure.

Variation of Compressive strength

The compressive strength of the concrete provides the idea of all the characteristics of concrete. It is the ability of a material or structure to carry loads on its surface without any crack or deflection. As seen from Table 3 and Fig-4, the Compressive strength of A3 (Plain cement+MWCNT's+Glass fibres) composite was high as compared to the Plain cement cubes. The high strength is due to the addition of MWCNT's and Glass Fibres. Concrete compressive strength for general construction varies from 15 MPa to 30 MPa and higher in commercial and Industrial structures.

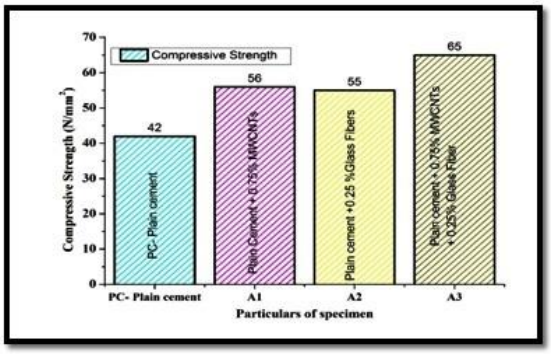


Figure-4. Showing variation of compressive strength by addition of nanoparticles.

SEM and EDS Analysis.

The purity of the produced materials in terms of their morphology and chemical composition was confirmed throughthe Energy Dispersive X-Ray Spectroscopy spectra. The chemical elements detected were C, Al, Si, S, and Ca. The major element found was Oxygenand Calcium. The

Oxygen helps in the reactions of MWCNT's and strength of the composite material is enhanced (Table 7, Fig 8).

Table 4.Table depicting Toughness Index

Specimen Particulars	Area under the curve (N-mm)	Toughness Index
PC	65.45	1
A1	95.5	1.46
A2	89.7	1.37
A3	164.7	2.52

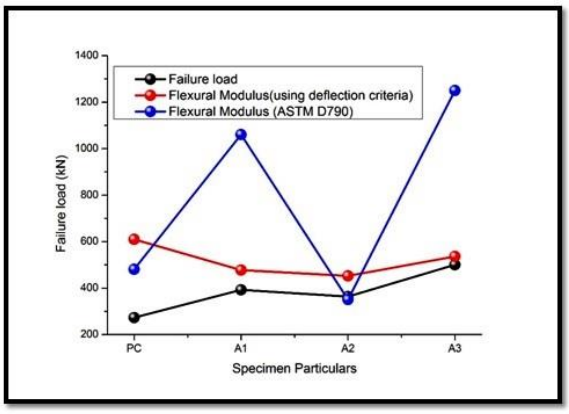


Figure-5. Graph showing the failure load for all compositions

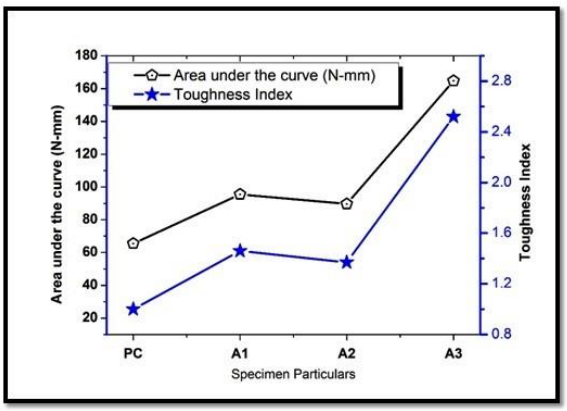


Figure-6. Graph depicting Area under the deflection curve and Toughness Index

SEM analysis was performed to assess the MWCNT's and Glass fibres dispersion quality and interaction of these nanoparticles in thecement paste. Considering the special characteristics of MWCNTs and glass fibres, such as its size and high aspect ratio (length to diameter) and also, these Glass Fibres with the C-S-H gel and hence the nanoparticles can be more efficiently distributed in very fine scales, compared to other reinforcement materials, which in turn enhances the strength of cement paste by bridging the cracks at initial stages of crack propagation in the matrix. MWCNT are more distinguishable at early ages in that the cement hydration products can be further attracted by the large surface energy of this nonmaterial over the curing time.

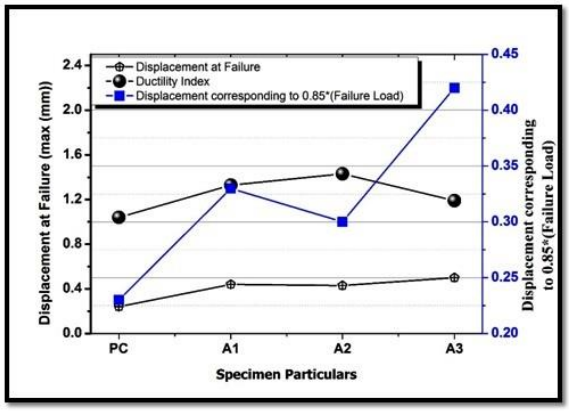


Figure-7. Graph depicting Ductility Index and displacement failure as in Table 5.

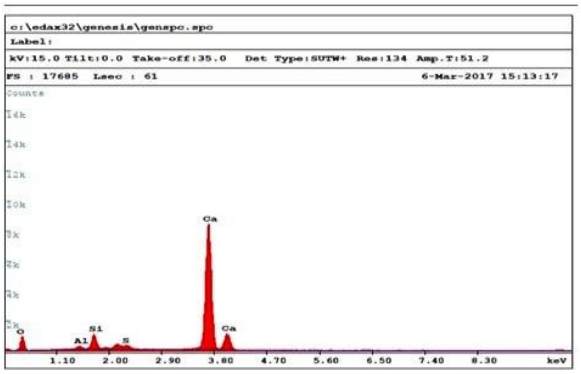


Fig-8. EDS Analysis for (PC) +MWCNT's+Glass fibres

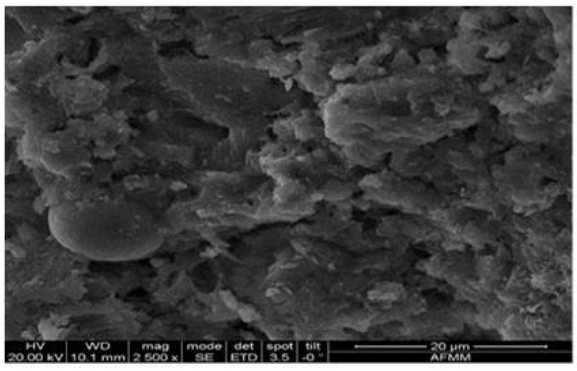


Figure-9. SEM image of cement paste modified with MWCNTs nano particles.

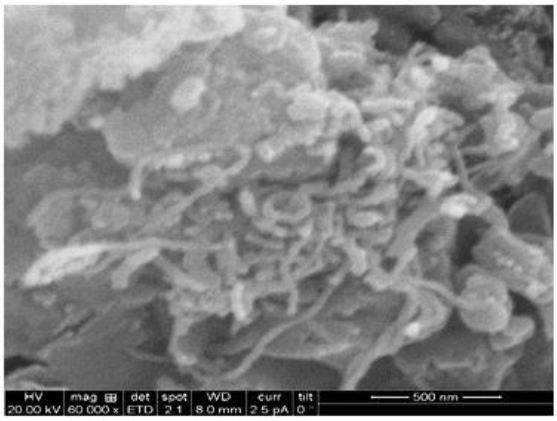


Fig-10. SEM Image showing hardened cement paste modified with Glass Fibres

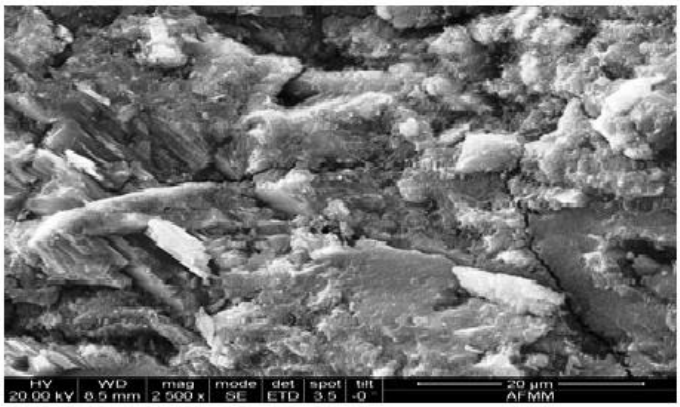


Fig-11. SEM Image showing hardened cement paste modified with MWCNTs and Glass Fibers

Table 5. Table depicting Ductility Index

Sr. No	Specimen Particulars		Failure Load(N)	Displacement Failure Δmax(mm)	Displacement corresponding to 0.85*(Failure Load) Δ0.85	Ductility Index = Δmax / Δ0.85
1	PC	Max	273	0.24	0.23	1.04
2	A1	Max	392	0.44	0.33	1.33
3	A2	Max	363	0.43	0.30	1.43
4	A3	Max	500	0.50	0.42	1.19

Table 6. Comparison of Compressive Strength of the composites subjected to Compression Test

Sl. No	Specimen type	Specimen specification	Compressive Strength. N/mm²	% increase or decrease
1	Plain cement	PC	42	_
2	PC+0.75 wt% MWCNTs	A1	56	33% increase
3	PC+0.25 wt% Glass Fibres	A2	50	19% increase
4	PC+0.75 wt% MWCNTs +0.25 wt% Glass Fibres	A3	65	55% increase

Table 7. Elemental composition from EDS Analysis for (PC+MWCNT's+Glass Fibers)

Element symbol	Element	% of element by weight of cement.(PC)	% of element by weight of cement.(0.75% MWCNT's)	% of element by weight of cement.(0.25% Glass Fibres)	% of element by weight ofcement.(PC+MWCNT's + Glass Fibers)
O	Oxygen	21.74	21.55	3.74	24.76
Al	Aluminium	2.1	1.19	1.68	2.94
Si	Silicon	12	4.11	1.82	11.35
S	Sulphur	1.9	1.43	62.71	2
Ca	Calcium	62	71.73	30.05	58.95
Total		100	100	100	100

The combination of C-S-H, MWCNT and Glass Fibres, which can be orientated in various directions, leads to the formation of a multiphase product that contributes to enhancing the mechanical characteristics of the cement paste. The pull out response of MWCNT facilitates the load transfer in the cement paste and improves the flexural strength. MWCNTs and glass Fibres should be uniformly dispersed within the cement paste to further enhance the strength characteristics in the matrix.

The formation of Carboxyl groups improves the bonding strength and as a result the overall strength of the nano composites increase. The strong Vander Waals forces lead to strong bonds between the cement matrix, Glass fibres and MWCNT's. The structure of MWCNT's as seen in EDS analysis, the formation of Carboxyl groups on the surface might have improved the bonding by inducing chemical reactions with hydraulic cementitiuous materials. The carboxyl groups in MWCNT's react with the C-S-H gel improving the bonding strength between the MWCNT's and the matrix.

Figure-9-11 shows the SEM image of hardened cement paste modified with MWCNT's, Glass fibres and combination of both the nano materials. In Fig-9,good pore filling has happened which might be the reason for the increase in strength and at the same time it shows clustering of MWCNT's which says that the dispersion might not have happened effectively. But, the enhancement of mechanical properties as observed in the results shows that the uniform dispersion must have happened. Fig-10 shows the SEM image of cement paste modified with Glass fibres. The figure says the dispersion is not up to mark as it shows clustering at some places.Fig-11 shows the SEM image of the cement paste modified with both MWCNT's and Glass Fibres. It shows the dispersion of both the nano materials i.e. MWCNT's and Glass Fibres. The uniform dispersion is seen at some places whereas the dispersion is not uniform at some other places. The uniform dispersion may be the result for good bonding and ultimately high flexural and compressive strength.

CONCLUSION

The presence of MWCNT's and Glass Fibres significantly improves the bonding within the cement composites and hence the propagation of micro cracks is prevented, the deflection hardening behavior is noticed to be increased with the increase of the MWCNTs content. It is noticed that the compressive strength also increases considerably with the addition of MWCNT's and Glass Fibers.

This is the best example of micro and nano level reinforcement in cement matrix. The maximum deflection was found to be 0.5mm with a maximum load of 500N. The flexural strength was observed to be 1250.50 N/mm2 as per ASTM D 790 which is 20% more than the flexural strength obtained with PC+MWCNT's and 60 to 70% more than that obtained with PC+ Glass Fibres. The flexural modulus as per deflection criteria is 535.94 N/mm2 which is 10 to 20% more than that obtained of PC+MWCNT's and PC+ Glass Fibres. The compressive strength of PC+0.75% MWCNt's+0.25% Glass fibres was found to be 65 N/mm2 which is greater than Plain cement and Plain cement+MWCNT's.

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